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Techniques for measuring the thermal conductivity of nanofluids: A review

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ABSTRACT

There has been a rapid progress in research activities concerning nanofluids since a large enhancement in their thermal conductivity has been reported a decade ago. While this extraordinary thermal conductivity of nanofluids deserves scientific investigation, the inconsistency and controversy of the results reported by different groups for identical nanofluids across the world raises fundamental doubts and poses a hindrance in the potential applications of nanofluids. This paper presents a critical review of the several techniques for the measurement of thermal conductivity of nanofluids employed by the researchers. Additionally, a detailed description of a unique thermal conductivity measurement device based on the thermal comparator principle, developed by the present authors has been described. Besides the principle of this measurement device, the constructional details have been elaborated. Finally, some suggestions have been made for improving the reliability of the measurement of thermal conductivity.

Contents

1. Introduction

Augmentation of heat transfer is one of the most difficult challenges faced by the thermal engineers continuously. With the advancement of technologies the demand for transferring heat at higher rates and efficiency from smaller areas or across a lower temperature difference is on perennial rise. Accordingly, over the decades different techniques for the enhancement of heat transfer have been suggested. Broadly these techniques may be classified between two categories – active and passive. Though the active techniques are more efficient and can be controlled better, they require auxiliary power and greater maintenance. On the other hand, passive techniques are self-sustained and have higher reliability due to the absence of auxiliary power sources, prime movers or moving components. Use of additives in heat transfer fluid for augmenting its heat transfer characteristics is a well known passive technique.

The thermal conductivity of most of the heat transfer fluids is orders of magnitude lower compared to solid metals (Fig. 1). The addition of micro- or milli-sized solid metal or metal oxide

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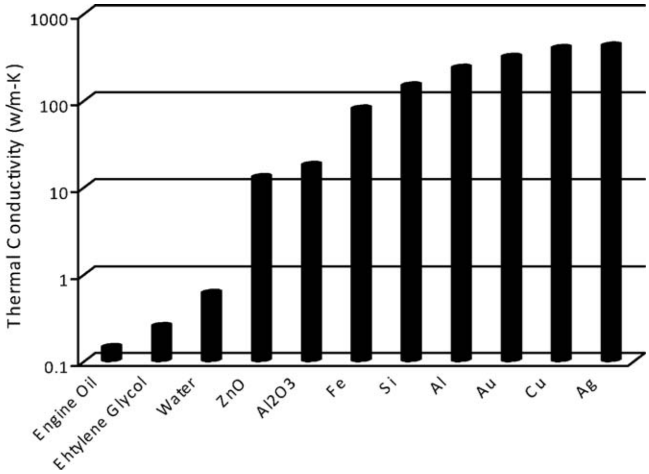

Fig. 1. Comparison of thermal conductivity of common liquids and solids.

particles to the base fluids shows an increment in the thermal conductivity of resultant fluids. But the presence of milli- or micro-sized particles in a fluid poses a number of problems. They do not form a stable solution and tend to settle down. Apart from the application in the field of heat transfer, nanofluids can also be synthesized for unique magnetic, electrical, chemical, and biological applications. They also cause erosion and clogging of the heat transfer channels. As a remedy to these problems it was proposed that fluids containing nanometer-sized particles can be a new class of engineered fluids offering exciting thermal properties to suit the heat transfer industry. The term nanofluid was proposed by Choi [1] in 1995 of Argonne National Laboratory, U.S.A. for such type of fluids.

Nanofluid can be defined as a fluid with nanoparticles suspended in it forming a stable colloid and constituting a quasi-single phase medium. One of the most important criteria of the nanoparticles dispersed in the nanofluids is that; at least one of their critical dimensions must be smaller than about 100 nm. The nanofluids not only show a substantially high thermal conductivity but also possess much better stability compared to any dispersion of micro-sized particles in a base fluid [2]. Due to their significantly improved thermal properties nanofluids could have potential applications in fields such as micro-electronics, transportation, manufacturing, and medical. When, a better utilization of energy and development of sustainable technology is the call of the day, nanofluids can play a very important role. Justifiably enough, nanofluids have attracted the attention of researchers from diverse fields. Volume of research work including a few review articles [3–7] has been published within a short span of time.

A review of the available literature shows that the increase in thermal conductivity of the nanofluids is highly anomalous. Addition of only a small volume percent of solids produces a dramatic increase in thermal conductivity [8–11]. It has been observed that even the addition of nano-sized ceramic particles in a base fluid enhances its thermal conductivity substantially [2,3,23,41,66]. The resultant thermal conductivity cannot be predicted from the rule of average. There are certain theories which derive the properties of two-phase mixtures from a more fundamental physics, for example, Maxwell's theory [12] and Hamilton and Crosser approach [13]. These formulations are also inadequate in predicting the thermal conductivity of nanofluids. Many of the researchers suggested altogether new mechanisms for the transport of thermal energy [14]. Such models met with only a partial success.

Till now, major volume of the work in the area of nanofluids has been dedicated to synthesis, characterization, and some applications in convective heat transfer and boiling. Synthesis of nanofluids can be done by two processes, namely, the one-step process (collecting freshly synthesized nanoparticles in the concerned medium) and the two-step process (dispersing nanoparticles in a chosen medium) [15–21]. The convective heat transfer studies executed by several researchers have shown appreciable increase in the heat transfer coefficient exhibited by nanofluids [22–29]. In pool boiling studies, there is a section of researchers showing enhancement in the boiling characteristics as a result of using nanofluid as the medium [30–32,36,37], whereas another group of researchers contradict such claims [33–35].

A multitude of investigations has reported different techniques for the estimation of thermal conductivity of nanofluids. These investigations have also presented a good deal of controversies. Experiments show that thermal conductivity of nanofluids depend on a large number of parameters namely, the chemical composition of the solid particle and the base fluid, particle concentration, particle shape and size, surfactants, etc. However, the correct trend of variation of the conductivity with these parameters is yet to be established. It has been found that temperature also affects the thermal conductivity of nanofluid. Several studies have been carried out to see the effect of temperature on the thermal conductivity of the nanofluids. Dependence of temperature on the thermal conductivity of CuO, Al_2O_3, TiO_2, ZnO [36–42] dispersed nanofluids have been studied by Mintsa et al. [38], Duangthongsuk and Wongwises [39], Vajjha and Das [40], Murshed et al. [41], and Yu et al. [42]. The increase in temperature increases the thermal conductivity of the nanofluids. However the real mechanism behind the increase has not yet been pointed out. It is not an exaggeration to state that the lack of reliable information regarding the conductivity of nanofluids act as a great impediment for its commercial exploitation.

Under the present scenario one needs to conduct careful experiments to measure the thermal conductivity with a well planned variation of the process parameters. Such experiments will help in establishing the effect of different variables on thermal conductivity. They will also be useful in validating different models proposed for the enhanced thermal conductivity of nanofluids.

The measurement of thermal conductivity of liquids is a challenging task. In general, Fourier's law of heat conduction is exploited for the measurement of thermal conductivity. In the simplest arrangement, one needs to establish a steady one-dimensional heat flow by the application of a known heat flux. Then by measuring the temperatures at two known locations along the direction of heat transmission one can estimate the thermal conductivity (Fig. 2) as follows

$$k = \frac{q/A}{\Delta T/L} \tag{1}$$

where q is the magnitude of heat transmission and ΔT is the temperature difference across length L and cross-sectional area A. In this scheme of measurement the greatest challenge lies in establishing a one-dimensional temperature field. Even in a homogenous isotropic solid, one needs to take special care and make appropriate design to achieve this. In case of liquids, additionally, any temperature gradient along the direction of gravity should be restricted. Otherwise, convection current will get established in the liquid and will produce a wrong estimate of the thermal conductivity.

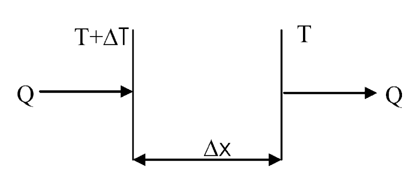

Fig. 2. Principle of heat conduction.

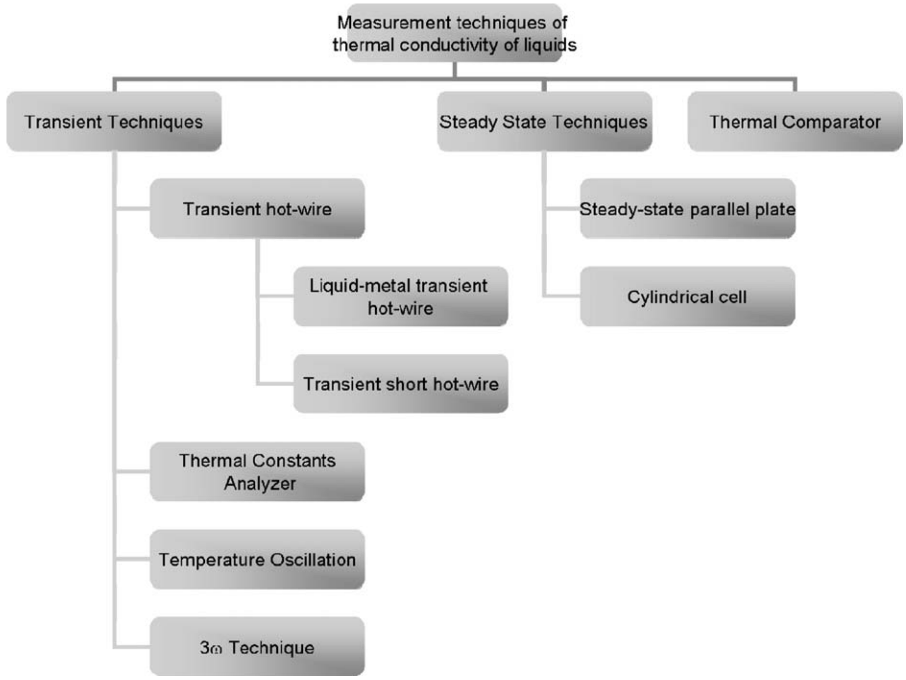

Fig. 3. Different thermal conductivity measurement techniques for nanofluids.

The occurrence of convection has to be suppressed in liquids to successfully measure their thermal conductivity. Fluids do not have a definite shape, size, and cross-sectional area which also make the thermal conductivity measurement a difficulty. In case of nanofluids, additionally the presence of suspended nanoparticles may cause a major problem for measurement of thermal conductivity as the homogeneity of the medium is to be maintained. The thermal conductivity of liquids can be successfully measured if the time taken to measure k is very small so that the convection current does not develop. Furthermore, instead of heating the liquid from below, heating it from above facilitates conduction of heat in a layer wise manner. Keeping these points in mind several techniques have been proposed to measure the thermal conductivity of nanofluids over the past few years. The most common techniques for the measurement of effective thermal conductivity of nanofluids are the transient hot-wire method [3], steady-state method [4], cylindrical cell method [5], temperature oscillation method [6], and 3-omega method [7] to name a few.

In this paper we present a review of each of the techniques for the measurement of thermal conductivity. The theory underlying each of the techniques, important features of the particular measurement, advantages and limitations have been discussed. A thermal comparator device based on the original idea of Powell [43] has been indigenously developed in our laboratory for the measurement of thermal conductivity of nanofluids. In this study we have also presented a detailed description of the comparator device along with some results. Finally, based on the present review a critical appraisal has been made for different techniques of measurement. This also includes suggestions which can make the measurements more reliable and usable.

2. Thermal conductivity measurement techniques for nanofluids

Over the years different techniques have been adopted for measuring the thermal conductivity of liquids. A number of such techniques have also been used for nanofluids. Fig. 3 provides a summary of the available measurement techniques. Out of all the techniques, the transient hot-wire method has been used most extensively. Based on the literature survey a relative popularity and frequency of use of each of the methods for the characterization of nanofluids has been presented in Fig. 4. Illustrations of the techniques given in Fig. 3 are given in the following sections.

2.1. Transient hot-wire technique

The transient hot-wire (THW) method was first suggested by Stalhane and Pyk (Horrocks and McLaughlin [44]) in 1931 to

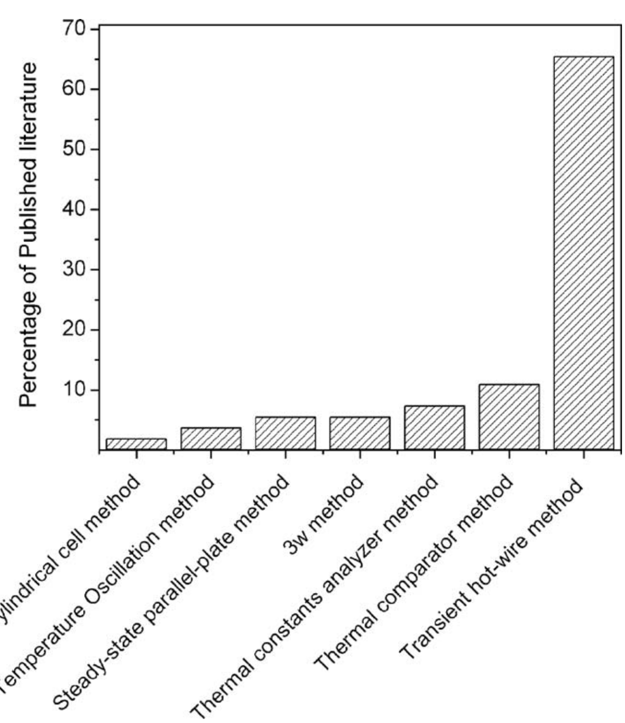

Fig. 4. Comparison of the thermal conductivity measurement techniques for nanofluids.

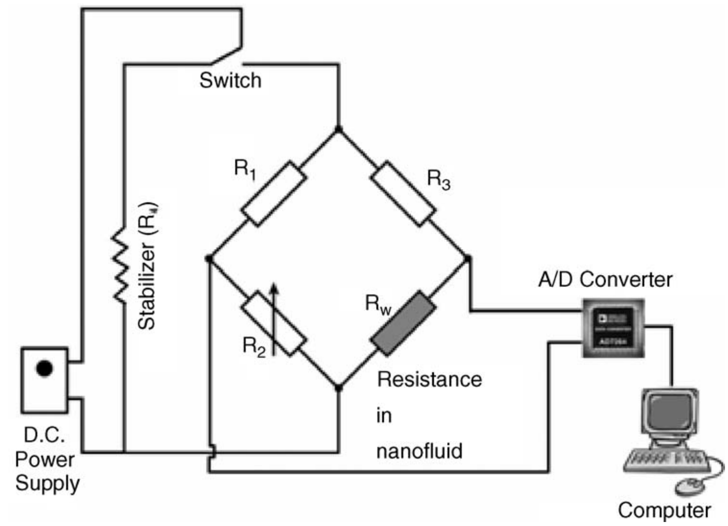

Fig. 5. Schematic of transient hot-wire experimental setup.

measure the absolute thermal conductivity of powders. Many researchers have modified the method to make it more accurate. There are several advantages for the TWH method. The most attractive advantage of this method for application to fluids is its capacity for experimentally eliminating the error due to natural convection. In addition, this method is very fast compared to other techniques. The conceptual design of the hot-wire apparatus is also simple compared to the arrangements needed for other techniques.

In this method, a platinum wire is used for the measurement. The wire is used both as a heater and as a thermometer. This method is based on the principle of measurement of temperature and time response of the wire subjected to an abrupt electrical pulse. Carslaw and Jaeger [45] modeled the temperature surrounding an infinite line heat source with constant heat output and zero mass, in an infinite medium. If heat at a constant rate, q is applied to an infinitely long and infinitely thin 'line' source, the temperature response of the source over time can be described by the equation:

$$\Delta T = -\frac{q}{4\pi k} Ei\left(\frac{-r^2}{4Dt}\right) \quad 0 < t < t_1 \tag{2}$$

where k is the thermal conductivity of the medium in which the line is buried, D is the thermal diffusivity of the medium, r is the distance from the line at which temperature is measured, and Ei is the exponential integral. Ei is defined in the following equation, and can be approximated by the series shown below [67]

$$-Ei(\alpha) = \int_{\alpha}^{\infty} \frac{1}{u} \exp(-u)\, du = -\beta - \ln\alpha - \frac{\alpha^2}{4} + \cdots \tag{3}$$

in which $\beta = 0.5772\ldots$ is Euler's constant and $\alpha = r^2/4Dt$.

The terms beyond $\ln\alpha$ in the series expansion of Ei become negligibly small for long times especially when r is small and D is large. So Eq. (3) can be approximated as

$$\Delta T \approx \frac{q}{4\pi k}\left[-\beta - \ln\left(\frac{r^2}{4Dt}\right)\right] = \frac{q}{4\pi k}\left[\ln t - \ln\left(\frac{r^2}{4DC_E}\right)\right] \tag{4}$$

where $C_E = \exp\beta$. Thus, after some delay, a graph of ΔT versus $\ln t$ becomes a straight line with slope equal to $q/4\pi k$. Since two points define a straight line, k can be computed from

$$k = \left[\frac{q}{4\pi(\Delta T_2 - \Delta T_1)}\right] \ln\left(\frac{t_2}{t_1}\right) \tag{5}$$

Standard instruments using this THW method has been manufactured over the years. In general, a transient hot-wire instrument consists of a probe which is to be inserted into the nanofluid for the measurement. A metallic wire functions as the probe which is used as a line heat source as well as a thermometer. A constant current is supplied to the wire to raise its temperature. The heat dissipated in the wire increases the temperature of the wire as well as that of the nanofluid. This temperature rise depends on the thermal conductivity of the nanofluid sample in which the hot-wire is inserted. Fig. 5 shows the schematic diagram of the arrangement which can be used for the measurement. Several researchers across the world have used transient hot-wire technique for the measurement of thermal conductivity of nanofluids. Some of them include Murshed et al. [2], Zhang et al. [46], Hong et al. [47], Kwak and Kim [48], Timofeeva et al. [49], He et al. [50], and Lee et al. [51]. Several variations of the transient hot-wire method, namely the liquid metal transient hot-wire technique and the transient short hot-wire have been used to measure the thermal conductivity of liquids.

The liquid metal transient hot-wire method is used for electrically conducting liquids. A mercury-filled glass capillary is suspended in the fluid or dispersion, with the glass capillary serving to insulate the mercury "hot-wire" from the electrically conducting fluid or dispersion [52]. The mercury wire forms one resistor in a Wheatstone bridge circuit and is heated when a constant voltage is applied to the bridge. The temperature rise of the wire is calculated from the change in the resistance of the mercury with time, obtained by measuring the voltage offset of the initially balanced Wheatstone bridge.

For measuring the thermal conductivity of highly corrosive liquids, such as molten carbonates, it is very difficult to keep the samples molten for such a long zone at a homogeneous initial temperature which is critical to obtain reasonable results. This disadvantage of TWH method can be solved by using a shorter probe (10 mm height) to measure the thermal conductivity of nanofluids using a smaller sample cell. The transient short hot-wire (SHW) technique is based on the numerical solution of two-dimensional transient heat conduction for a short wire with the same length-to-diameter ratio and boundary conditions as those used in the actual measurements [53].

2.2. Thermal constants analyzer technique

The thermal constants analyzer utilizes the transient plane source (TPS) theory to calculate the thermal conductivity of

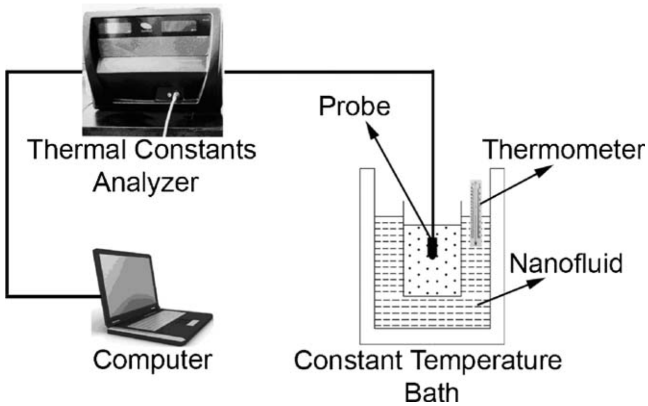

Fig. 6. Schematic diagram of the experimental setup for transient plate source method.

nanofluid. In this method, the TPS element behaves both as the temperature sensor and the heat source. The TPS method uses the Fourier law of heat conduction as its fundamental principle for measuring the thermal conductivity, just like the THW method. Advantages of using this method are (a) the measurements are fast, (b) samples having wide range of thermal conductivities (from 0.02 to 200 W/m K) can be measured, (c) no sample preparation is required, and (d) sample size can be flexible [54].

The experimental setup (shown in Fig. 6) comprises of thermal constants analyzer, a vessel, a constant temperature bath, and a thermometer. The probe of the thermal constants analyzer is immersed vertically in the vessel containing the nanofluid. The vessel is placed in the constant temperature bath and the thermometer is immersed in the vessel to measure the temperature of the nanofluid. The thermal conductivity of the nanofluid is determined by measuring the resistance of the probe. The probe consists of an electrically conducting thin foil of a typical pattern which is sandwiched inside an insulating layer, as shown in Fig. 7 [20]. When a constant electric power is supplied to the probe, the temperature rise of the probe, $\Delta T(\tau)$, can be measured by the probe resistance with time, $R_p(\tau)$:

$$\Delta T(\tau) = \frac{1}{\alpha}\left[\frac{R_p(\tau)}{R_0} - 1\right] \tag{6}$$

where α is the temperature coefficient of the electric resistance, R_0 is the electric resistance of the probe when $\tau = 0$, and τ is the variable on the time of electrification and be defined as:

$$\tau = \sqrt{\frac{tR}{r_p^2}} \tag{7}$$

where t is the measuring time, R is the thermal diffusivity of fluid, and r_p is the radius of the probe.

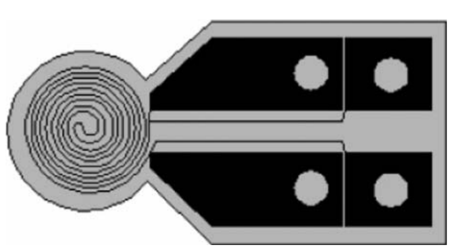

Fig. 7. Schematic diagram of TPS sensor.

According to Fourier Law of heat conduction, if no natural convection of a fluid occurs, $\Delta T(\tau)$ can also be calculated as [55]:

$$\Delta T(\tau) = \frac{W}{\pi^{1.5} r_p k} D(\tau) \tag{8}$$

$$D(\tau) = \int_0^{\tau} d\sigma(\sigma^{-2}) \int_0^1 \nu\, d\nu \int_0^1 u\, du \times \exp\left(\frac{-u^2 - \nu^2}{4\sigma^2}\right) I_0\left(\frac{u\nu}{2\sigma^2}\right) \tag{9}$$

where W is the electric power supplied to the probe, k is the thermal conductivity of fluid, I_0 is a modified Bessel function, and $D(\tau)$ is a geometric function.

In the absence of any natural convection, by fitting the experimental data to the straight line given by Eq. (8), the thermal conductivity of the fluid can be obtained from the slope of the line $W/(\pi^{1.5} r_p k)$. If natural convection of fluid occurs, the thermal conductivity calculated by Eq. (8) will vary with $D(\tau)$, and the result is not correct. In this case, the thermal constants analyzer can automatically give an alarm to avoid using the unreliable result. In order to avoid the happening of natural convection, the parameters of the analyzer should be controlled properly.

The thermal constants analyzer has been used by Zhu et al. [54] and Jiang et al. [56] for measuring the thermal conductivity of nanofluids.

2.3. Steady-state parallel-plate method

Based on steady-state heat conduction various design of test cells can be constructed for the measurement of thermal conductivity of liquids. To facilitate the heat transfer predominantly in one direction either parallel-plate type or concentric cylindrical cell type test facilities are preferred. The apparatus for the steady-state parallel-plate method can be constructed on the basis of the design by Challoner and Powell [4]. A schematic diagram of the experimental set up is shown in Fig. 8 where a small volume of the fluid sample is placed between two parallel round pure copper plates. A detailed description of the set up has been given by Wang et al. [57]. They have used this method for measuring the thermal conductivity of alumina and copper oxide based nanofluids. In this method, two important parameters are to be carefully controlled. One needs to accurately measure the temperature increase in each thermocouple. The difference in temperature readings need to be minimized when the thermocouples are at the same temperature. As the total heat supplied by the main heater flows through the liquid between the upper and lower copper plates, the overall thermal conductivity across the two copper plates, including the effect of the glass spacers, can be

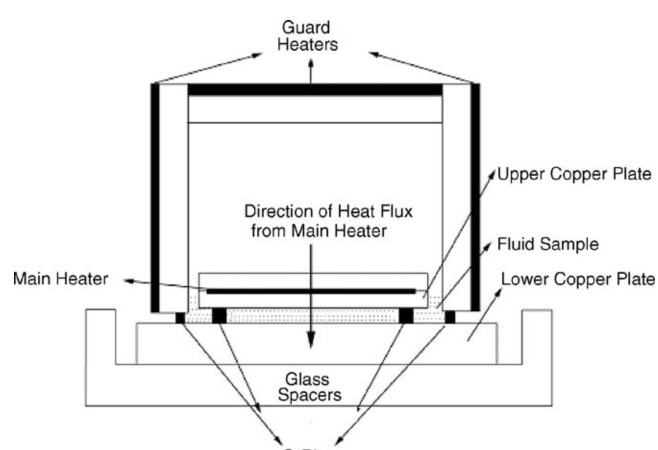

Fig. 8. Experimental set up for steady-state parallel-plate method.

calculated from the one-dimensional heat conduction equation relating the power $\dot{q}$ of the main heater, the temperature difference ΔT between the two copper plates, and the geometry of the liquid cell as

$$k = \frac{\dot{q}L_g}{S\Delta T} \tag{10}$$

where L_g is the thickness of the glass spacer between the two copper plates and S is the cross-sectional area of the top copper plate. The thermal conductivity of the liquid can be calculated as

$$k_e = \frac{kS - k_g S_g}{S - S_g} \tag{11}$$

where k_g, S, and S_g are the thermal conductivity, cross-sectional area of the top copper plate, and the total cross-sectional area of the glass spacers, respectively. In this method, it has to be ensured that there is no heat loss from the fluid to the surrounding. To take care of this, guard heaters (shown in Fig. 8) are used to maintain a constant temperature of the fluid. The guard heaters are heated to a temperature same as that of the fluid. If the fluid and the guard heater temperature are equal, then there will be no heat radiated to the surroundings from the fluid.

2.4. Cylindrical cell method

Cylindrical cell method is one of the most common steady-state methods used for the measurement of thermal conductivity of fluids. In this method the nanofluid whose thermal conductivity is to be measured fills the annular space between two concentric cylinders. Kart and Kayfeci [5] has given a detailed description of the equipment. A brief description is as follows. The equipment (shown in Fig. 9) consists of a coaxial inner cylinder (made of copper) and outer cylinder (made of galvanize). An electrical heater is placed inside the inner cylinder and the front and back sides of the equipment are insulated to nullify the heat loss during the measurement. During the experiment, heat flows in the radial direction outwards through the test liquid, filled in the annular gap, to the cooling water. Two calibrated Fe–Constantan thermocouples are used to measure the outer surface temperature of the glass tube (T_i) and the inner cylinder (T_o). The thermocouples are positioned in the middle of test section and connected to a multi channel digital read out with an accuracy of 0.1 °C. The required measurements for the calculation of the thermal conductivity are the T_i and T_o temperatures, adjusted voltage and current of the heater.

Using Fourier's equation in cylindrical co-ordinates, the thermal conductivity of nanofluid in the gap can be calculated by the equation,

$$k = \frac{\ln(r_2/r_1)}{2\pi L[(\Delta T/\dot{Q}_e) - (\ln(r_3/r_2)/2\pi L k_c)]} \tag{12}$$

where the heat input, $\dot{Q}_e$ (W) was calculated from measurement of the current and voltage through the heater ($\dot{Q}_e$), ΔT is temperature difference between T_i (°C) and T_o (°C), k_c is thermal conductivity of

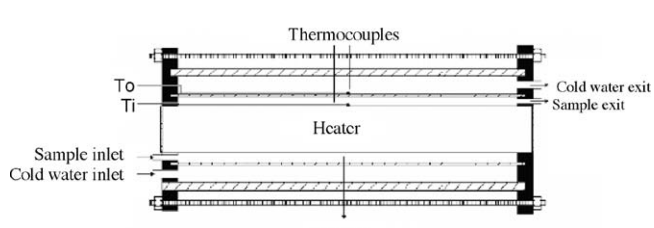

Fig. 9. Cross-section of the cylindrical cell equipment.

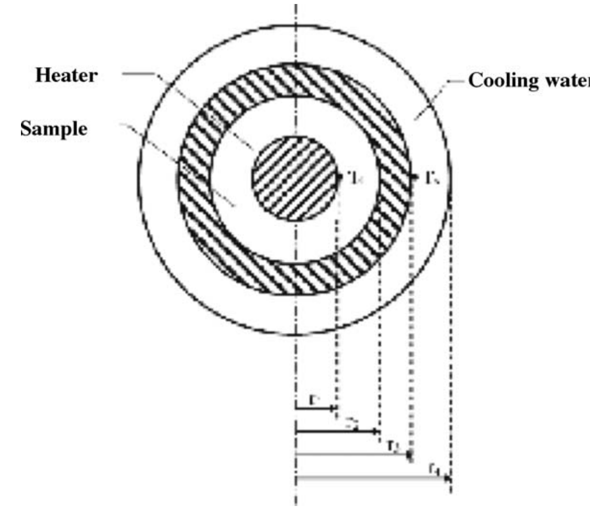

Fig. 10. Thermal resistances related to the cylindrical cell equipment.

copper (W/mK), L is length of cylinders (m), r_1 is outer radius of the glass tube (m), and r_2, r_3 are inner and outer radius of the inner cylinder (m) respectively, as shown in Fig. 10.

2.5. Temperature oscillation technique

This method measures the temperature response of the nanofluid when a temperature oscillation or heat flux is imposed. The measured temperature response of the nanofluid is the result of averaged or localized thermal conductivity in the direction of nanofluid chamber height. The experimental method used here is based on the oscillation method proposed by Roetzel et al. [6] and further developed by Czarnetski and Roetzel [58]. The principle of thermal conductivity measurement has been described by Das et al. [59], who have used this technique to measure the thermal conductivity of nanofluids comprising of Al_2O_3 and CuO nanoparticles dispersed in water. The experimental setup (shown in Fig. 11) requires a specially fabricated test cell (1) which is cooled by cooling water (2) on both the ends, coming from a thermostatic bath (3). Electrical connection provides power to the Peltier element (4). The temperatures are measured in the test section (shown in Fig. 12) through a number of thermocouples and these responses are amplified with amplifier (5) followed by a filter which is finally fed to the data acquisition system (6) comprising of a card for logging the measured data. The data logger is in turn connected to a computer with proper software (7) for online display which is required to assess the steady oscillation and for recording data.

From the principle of measurement it is evident that thermal diffusivity of the fluid can be measured very accurately by considering amplitude attenuating of thermal oscillation from the boundary (fluid reference material interface) to the center of the fluid. However for direct measurement of thermal conductivity one has to consider the attenuation at the reference material as well. Since the reference material has been worked upon and the micro-cracks and inhomogenity of material brings out uncertainty in its thermal conductivity value, direct evaluation of thermal conductivity of fluid is less accurate. Hence, in the present measurement the value of thermal diffusivity for the nanofluid is evaluated from experiment. Subsequently the density has been measured and specific heat is calculated from handbook, as

$$C_{p,nf} = \frac{m_s C_{p,s} + m_w C_{p,w}}{m_s + m_w} \tag{13}$$

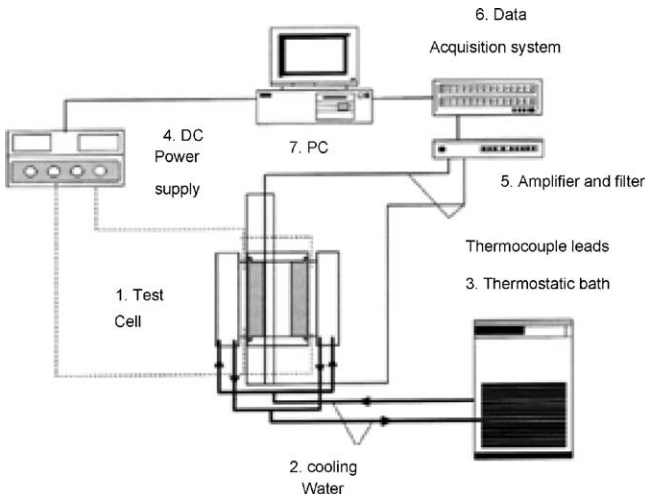

Fig. 11. Schematic of experimental set up for temperature oscillation technique.

The thermal conductivity can be calculated as,

$$k_{nf} = \alpha_{nf}\rho_{nf}C_{p,nf} \tag{14}$$

2.6. 3ω method

Similar to hot-wire technique, the 3ω method uses a radial flow of heat from a single element which acts both as the heater and the thermometer. The use of the temperature oscillation instead of the time dependent response is the major difference. A sinusoidal current at frequency ω passes through the metal wire and generates a heat wave at frequency 2ω, which is deduced by the voltage component at frequency 3ω. According to Cahill [7], the exact solution at a distance $r = (x^2 + y^2)^{1/2}$ from an infinitely narrow line-source of heat on the surface of an infinite half-volume is given by,

$$\Delta T(r) = \frac{P}{l\pi k} K_0(qr) \tag{15}$$

where k is the thermal conductivity of the half-infinite volume, and P/l is the amplitude of the power per unit length generated at a frequency 2ω in the line source of heat. The factor 2 is because a current at frequency ω produces joule heating at frequency 2ω. K_0 is the zeroth-order modified Bessel function.

The magnitude of the complex quantity,

$$\frac{1}{q} = \left(\frac{D}{i2\omega}\right)^{1/2} \tag{16}$$

is the wavelength of the diffusive thermal well or the thermal penetration depth. D is the thermal diffusivity of the material.

In the limit, $|qr| \ll 1$

$$\Delta T(r) = \frac{P}{l\pi k}\left(\frac{1}{2}\ln\frac{D}{r^2} + \ln 2 - 0.5772 - \frac{1}{2}\ln 2\omega - \frac{i\pi}{4}\right) \tag{17}$$

Either the real and imaginary part of temperature oscillations (Eq. (6)) can be used to find the thermal conductivity. The temperature oscillation and the heat generation rate are related by,

$$\Delta T = \frac{P}{l\pi k}\int_0^\infty \frac{\sin^2 kb}{(kb)^2 (k^2 + q^2)^{1/2}}\, dk \tag{18}$$

The 3ω device is fabricated by metal deposition and patterning. The device is connected to metal heaters by electrical wires. A well is created around the heater which contains the nanofluid. The microdevice is placed inside a temperature-controlled cryostat. This method is mostly used for the measurement of temperature dependent thermal conductivity of nanofluids. Oh et al. [60] used this method to measure the thermal conductivity of Al_2O_3 dispersed in DI and ethylene glycol at room temperature.

2.6.1. Thermal comparator method

Powell [43] described a unique technique for the measurement of thermal conductivity. This indirect technique is based on the principle of thermal comparator. The probe used in this technique only needs a point contact with the sample whose conductivity is to be measured. Further, the measurement is almost instantaneous. These features render this technique very suitable for the measurement of thermal conductivity of different liquids. Following the original concept of Powell [43], a thermal comparator setup was indigenously developed by the present research group to measure the thermal conductivity of nanofluids. It is a well-known principle that when two materials at different temperatures are brought in contact over a small area, heat transfer takes place from the hotter to the colder body. As a result, an intermediate temperature is very quickly attained at the point of contact. The contact temperature depends on thermal conductivity of two materials. Thermocouples are used to measure the voltage proportional to the temperature difference between the thermocouple probe tip and a reference located within the heated probe is measured. Using samples of known thermal conductivity, a calibration curve is prepared. Using the calibration curve the thermal conductivity of the unknown samples can be estimated readily.

The setup developed in-house consists of a metallic copper probe, a temperature-controlled heating coil, a direct current micro-voltmeter, and a voltage stabilizer (shown in Fig. 13). The probe is most the important part of the setup as the success of the method depends on the heat flow from the probe to test material through very small area of contact. The probe is made of copper. A heater is placed around the probe to compensate for the heat loss from the probe and to maintain a constant temperature difference between the probe and the sample. A copper–constantan (T type) thermocouple is connected between the probe and the test material. This gives the initial temperature difference between the two. This temperature difference is to be kept constant throughout the experiment. Major part of the heat transfer occurs under steady-state condition. There should be a direct relation between

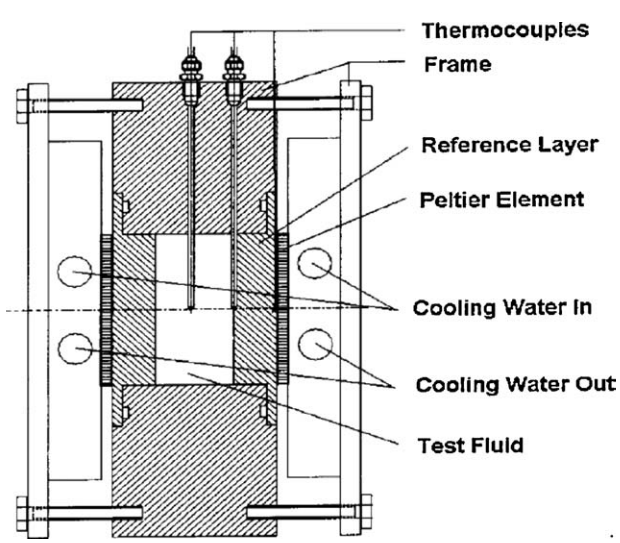

Fig. 12. Test cell construction.

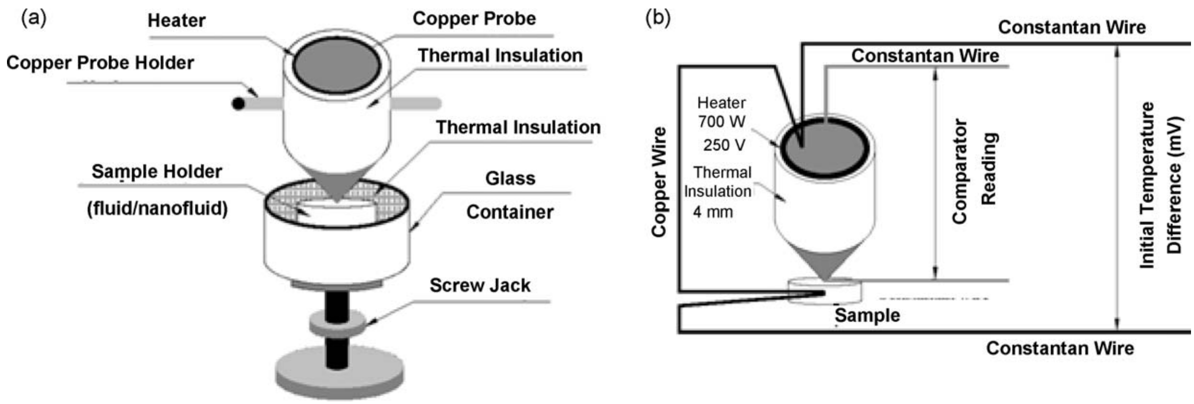

Fig. 13. Thermal conductivity measurements based on the thermal comparator method (b) the principle for recording differential thermo-emf using (a).

thermal conductivity (k) and observed differential emf (V). The test is made under conditions that ensure that k of the test material is the only variable. A copper-constantan thermocouple (T type) is located at the top of the steel block as a reference junction. The sample or liquid is placed on a height-adjustable platform that can be move up or down to ensure the point contact between the probe and the sample liquid the platform is slightly raised. For comparative testing, contact between the probe tip and the sample should be a point contact. When an electrically heated source is brought in contact with the sample (fluid) surface, the thermocouple attached to the probe tip senses the temperature. The generated thermo-emf in the circuit covering the probe assembly, sample (nanofluid) and base is recorded. The thermo-emf is proportional to the temperature difference between the thermocouple probe-tip and reference.

The contact area of a sphere on a plane is known to be small for moderate loading: indeed, anticipating a result derived later, the diameter of the contact area is of the order of 0.01 cm. Hence the diameter of the sphere is greater by approaching two orders of magnitude, and under these circumstances the system may be regarded as that of one semi-infinite solid in contact with another over a small circular area. This approximation is valid when the conductivity of the sphere has medium to high values.

In solving the problem, heat flow is considered between two semi-infinite bodies $0 < z < \infty$, and $-\infty < z < 0$ having thermal conductivities k_1 and k_2, and initial temperatures V_0 and 0 respectively. The two regions are in contact over a radius of r_1 (shown in Fig. 14). Considering appropriate equations and boundary conditions for solving the problem it can be derived that the temperature of region 1, V_1, is given as:

$$V_1 = V_0 - \frac{2V_0 k_2}{\pi(k_1 + k_2)} \int_0^{\infty} e^{-\lambda z} (\sin \lambda r_1) J_0(\lambda r) \frac{d\lambda}{\lambda} \tag{19}$$

where k_1 and k_2 are the thermal conductivities of region 1 and region 2 respectively, and $J_0(\lambda r)$ is the zero order Bessel function. For $z = 0$, and $r < r_1$, Eq. (19) reduces to

$$V_1(0) = \frac{V_0 k_1}{k_1 + k_2} \tag{20}$$

The derivation and the mathematical details of the function have been described elsewhere [62]. The effect of the transient term of heat conduction has also been considered. It has been found out that the contribution of this term is very insignificant compared to the effect of the steady state [61]. Thus the comparator can be considered as a steady-state device. From Eq. (20), it can be seen that the temperature of the contact region is independent of the contact radius and is uniform over the contact area. Eq. (20) states that if the contact temperature, $V_1(0)$, initial temperature of one region, V_0, and thermal conductivity of one substance, k_1 is known, then the thermal conductivity of the other substance, k_2 can be easily determined. The principle of the thermal comparator developed by Powell [43,61] has been used for measuring the thermal conductivity of several materials [62,63,64]. In the thermal comparator device developed and fabricated by our group, the temperatures $V_1(0)$ and V_0 are measured in terms of voltage output. So the contact voltage and the thermal conductivity of the copper probe being known quantities, the thermal conductivity of the fluid, which is brought into contact with the probe for a very short period of time, can be determined.

A calibration curve (shown in Fig. 15) is obtained by recording the thermo-emf of various standard fluids using the thermal comparator. Regression analysis of the variation in thermal conductivity (k) as a function of comparator reading (x) yields a polynomial to correlate x (in V) with k (in W/m-K) as follows:

$$k = a + bx + cx^2 \tag{21}$$

where a, b, and c are the regression coefficients with values $a = 2.9 \times 10^{-1}$ W/m-K, $b = -2.5 \times 10^{-3}$ W/m-K/V, and $a = 8.1 \times 10^{-6}$ W/m-K/V^2.

Initial measurements were carried out using water, ethylene glycol, liquid paraffin and carbon tetrachloride to obtain the calibration curve. The calibration was subsequently validated using two other common liquids, toluene and benzene. The comparator readings for toluene (218.2 μV) and benzene (221.1 μV), when converted to the corresponding thermal conductivity using Eq. (21), yielded values (0.137 and 0.140 W/m-K), which were within ±3% deviation from the corresponding standard

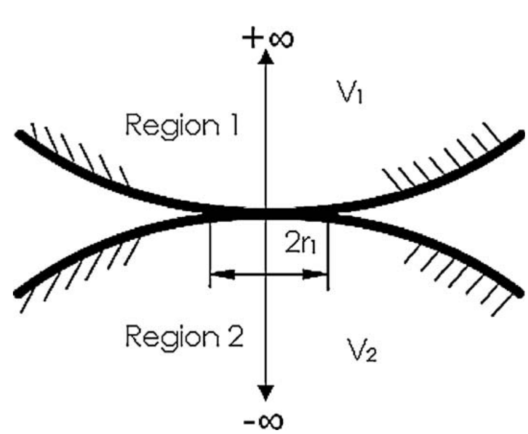

Fig. 14. Two semi-infinite regions in contact over a radius r_1 with temperatures V_1 and V_2 respectively.

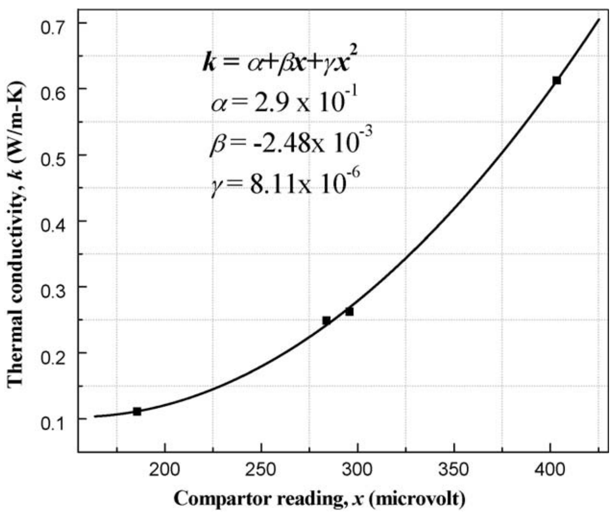

Fig. 15. Calibration curve to convert comparator reading (x) into thermal conductivity (k) of fluid/nanofluid (a, b, c are regression constants).

k values of toluene (0.133 W/m-K) and benzene (0.144 W/m-K) at a comparable temperature (300 K) [65]. This small deviation may arise due to the impurity (water) present in the fluids. Thus, Fig. 15 provides a reasonably accurate calibration to convert the experimental values of x (comparator reading) into the corresponding k (thermal conductivity) of the specific fluid or nanofluid.

The measurement procedure involves a few steps as follows. The probe is first heated by the coil wound on its outer surface, and kept for a period till the initial temperature difference between the probe and the test liquid is around to 25 °C. After the initial temperature difference is attained, the test liquid is taken in a glass cell and placed on the adjustable platform that is subsequently raised to make a point contact with the probe tip. As already pointed out that key to precise measurement in this setup lies in ensuring a point contact between the liquid surface and copper probe.

The major and important precautions to be exercised during measurement are:

1. The initial temperature difference between probe and sample kept a constant during the test.

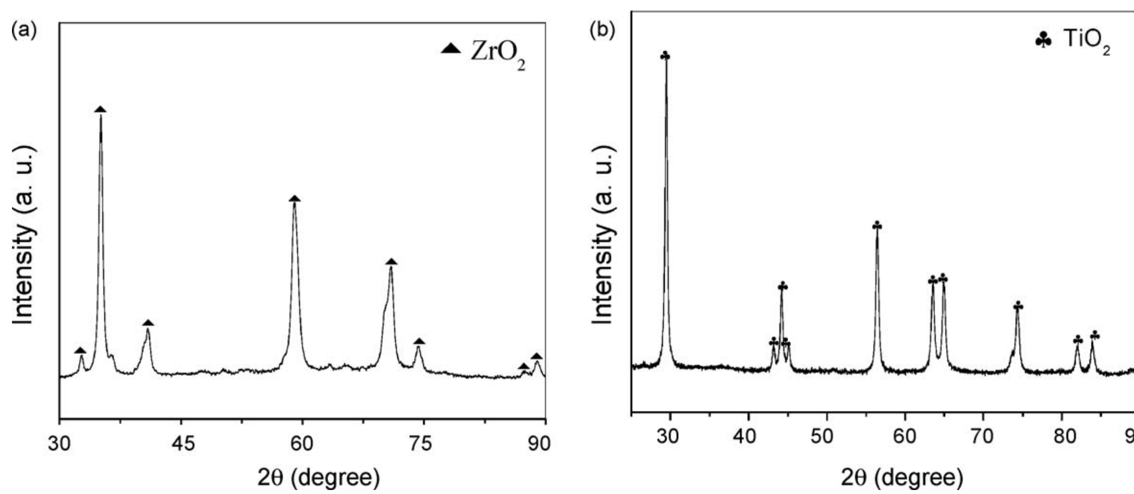

Fig. 16. XRD pattern of (a) ZrO_2 and (b) TiO_2 as received nano-ceramic particles.

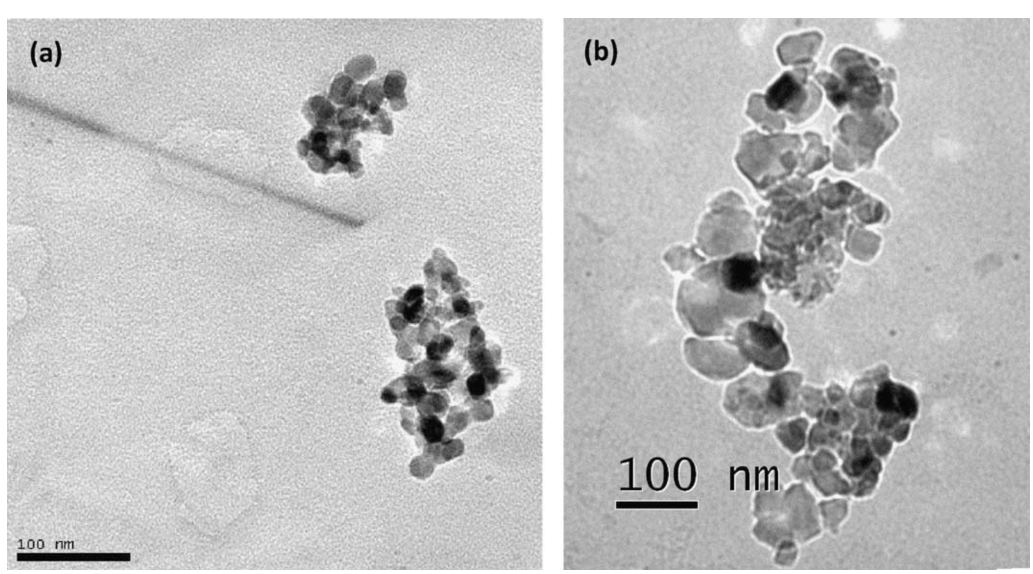

Fig. 17. TEM micrographs showing (a) ZrO_2 and (b) TiO_2 particles dispersed in water.

2. The contact between probe and sample should be a point contact.

3. Experimental results

3.1. Characterization of nanoparticles and preparation of nanofluids

In this study nano-ceramic based nanofluids were prepared by dispersing zirconia (ZrO_2) and titania (TiO_2) in base fluid water and ethylene glycol. Characterization of the nanoparticles was done by X-ray diffraction (XRD) for phase identification and crystallite size determination (Fig. 16). Selected samples were examined under transmission electron microscope (TEM) to study the size, dispersion and morphology of nanoparticles in the base fluid.

Fig. 17 shows the morphology and size distribution of ZrO_2 and TiO_2 nanoparticles used in this study. It is evident from the figure that the nanoparticles are indeed near spherical in shape having a tendency of clustering or agglomeration and non-uniform dispersion prior to de-agglomeration by ultrasonic vibration and magnetic stirring.

3.2. Thermal conductivity measurement

The thermal conductivity of the nanofluids has been measured the thermal comparator method. Fig. 18 shows the variation of thermal conductivity ratio for nanofluids (both ZrO_2 and TiO_2 nanoparticles dispersed in base fluid water and ethylene glycol) versus the nanoparticle volume percent. The results show significant increase in the thermal conductivity ratio with increase in volume percent for both ZrO_2 and TiO_2 based nanofluids. The increase for ethylene glycol based nanofluids in both cases is more compared to water based nanofluids as the former is a more viscous fluid. In terms of stability also the ethylene glycol based nanofluids are more stable than the water based nanofluids. The increase in thermal conductivity ratio is almost linear with increase in volume percent and reaches about 200% for ethylene glycol based ZrO_2 nanofluids.

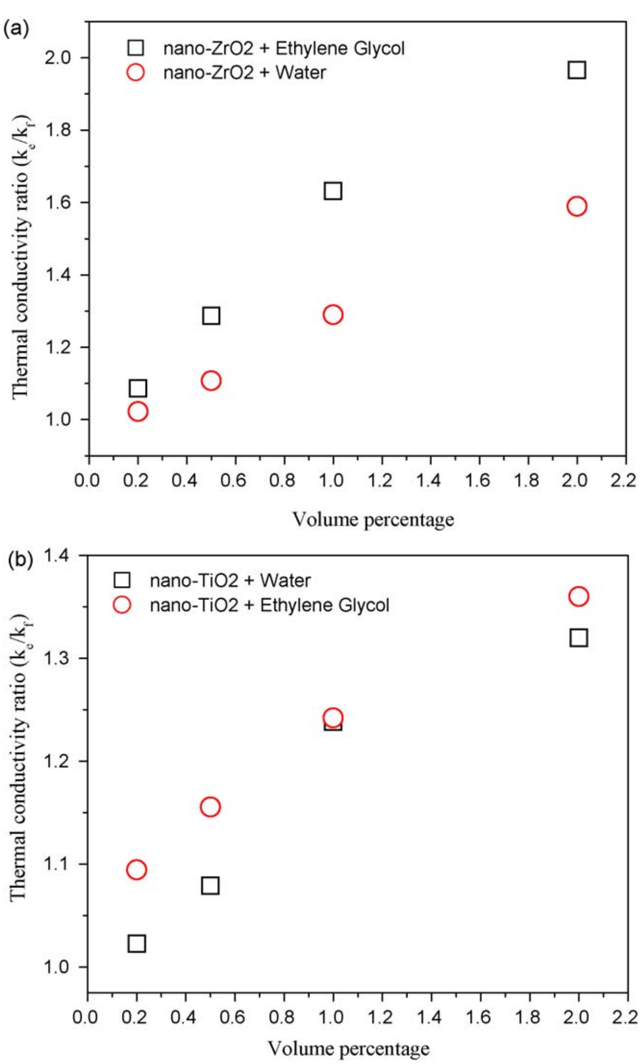

Fig. 18. Thermal conductivity ratio versus volume percentage for (a) ZrO_2 and (b) TiO_2 based nanofluids.

4. Conclusion

We have presented a brief review of the different techniques for the measurement of thermal conductivity of nanofluids available in literature. The review shows that the most commonly used technique for measuring thermal conductivity is the transient hot-wire technique. This measurement technique has gained popularity because of the fact that the thermal conductivity of the liquid can be measured instantaneously with a good level of accuracy and repeatability. From the literature survey a few suggestions may be pointed out.

- Some of the research groups have found anomalous increase in the thermal conductivity of nanofluids compared to the base fluids. In most of the cases, the thermal conductivity of the nanofluids has been measured using a single measurement technique. If the measurements can be done by a second method, the reliability of the data could be validated. This could explain the repeatability of the experiments as well.
- Though some amount [11,59,66,68–70] of work has been carried out to find out the effect of temperature on the thermal conductivity of nanofluids, all the research groups has performed the experiments starting from the room temperature. No work has yet been reported with experiments dealing the measurement of thermal conductivity at low (sub-zero) range of temperatures. The behavior of the thermal conductivity at low temperatures are yet to be found out and can point a new direction in this field of research.
- The behavior of the thermal conductivity of nanofluids with aging has not yet been reported. As the stability of nanofluids has been always a challenging task in this field of research, the study of the thermal conductivity with time of the nanofluids may show some unique feature that has not yet been reported.

In this study, a detailed description of an indigenously developed thermal comparator device developed by the present research group has been presented. We have prepared ethylene glycol and water based nano-ceramic (ZrO_2 and TiO_2) nanofluids using the two-step or physical preparation method. The effective thermal conductivity of the nanofluids has been measured in comparison to the base fluid with increase in volume percentage. The results show significant increase in thermal conductivity ratio with increase in volume percent. Nano-ZrO_2 shows the highest increase (nearly 2 times) when dispersed in ethylene glycol. The results are promising for development of nanofluids for thermal engineering applications.

Acknowledgements

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Appendix A. Nomenclature

k	Thermal conductivity
q	Magnitude of heat transmission
ΔT	Temperature difference across length and cross section
L	Length of the material
A	Cross-sectional area
D	Thermal diffusivity of the medium
R	Distance from the line at which temperature is measured
Ei	Exponential integral
β	Euler's constant
A	Temperature coefficient of the electric resistance
R_0	Initial electric resistance of the probe
T	Measuring time
R	Thermal diffusivity of fluid
r_p	Radius of the probe
W	Electric power supplied to the probe
I_0	Modified Bessel function
$D(\tau)$	Geometric function
$\dot{q}$	Power supplied to the main heater
L_g	Thickness of the glass spacer between the two copper plates
S	Cross-sectional area of the top copper plate
k_g	Thermal conductivity of the top copper plate
S_g	Total cross-sectional area of the glass spacers
$\dot{Q}_e$	Heat input by the central heater
k_c	Thermal conductivity of copper
r_1	Outer radius of the glass tube
r_2	Inner radius of the inner cylinder
r_3	Outer radius of the inner cylinder
ω	Angular frequency
P/l	Amplitude of the power per unit length generated at a frequency 2ω
K_0	Zeroth-order modified Bessel function
V	Differential emf
k_1	Thermal conductivities of region 1
k_2	Thermal conductivities of region 2
$J_0(\lambda r)$	Zeroth order Bessel function
$V_1(0)$	Contact temperature
a, b, and c	Regression coefficients

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